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THE INTERACTION OF IMPURITY ATOMS WITH DISLOCATIONS IN IRON

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ABSTRACT. The effect of the use of heat to release dislocations on the mechanical properties of iron, particularly the yield point, is investigated. Rapid cooling from different temperatures is used to fix the dislocations not locked by impurity atoms.

Locking dislocations with "atmospheres" of impurity atoms is considered an /216* important hardening mechanism [1]. Yet virtually no study has been made of this question experimentally, if one disregards the special field of deformation aging.

Iron with interstitial atoms of nitrogen and carbon is of particular interest /217 because theoretically, at least, the energy of interaction in these alloys should be high.

The "atmospheres" at the dislocations should dissipate at elevated temperatures, and it is this, presumably, that is associated with the disappearance of a yield stage for low-carbon iron at temperatures above ~ 300-400°C [2], as well as absence of a yield stage in iron hardened at 650-700°C. However, there still are no direct experiments proving the dissipation of "atmospheres."

This paper deals with the task of using rapid cooling from different temperatures to fix the dislocation not locked by impurity atoms, and to investigate the effect of the use of heat to release dislocations, on the mechanical properties of iron, particularly on the yield point.

The dependence of the low-temperature background of the internal friction on the amplitude can be measured to find out whether or not there is a link between dislocations and impurity atoms. Basic to the theories that have evolved as to this dependence is the fact that dislocations are freer the greater the dependence of the background on the amplitude [3].

Iron containing nitrogen and carbon totaling ~ 0.0002 percent by weight was prepared for the investigations. Pure iron was selected for the following reasons: (1) the fewer the impurity atoms, the easier it is to retain free

* Numbers in the margin indicate pagination in the foreign text.

dislocations during hardening; (2) the solubility of the interstitial atoms in the α-Fe lattice should remain constant at different temperatures if the process of unlocking the dislocations by using heat is not to be complicated by the processes involved in the liberation of carbides and nitrides.

Decarbonized iron power, containing 0.001 percent carbon, was pressed into briquettes and these were remelted in a vacuum induction furnance. This ingot then was made into a rod which then was used as an electrode in a zone vacuum furnace with a consumable electrode. The metal obtained by zone remelting was wire-drawn at ambient temperature to a diameter of 0.75 mm. We shall designate this as material A. The chemical composition of A iron, in percentages, was Al 0.017, Mn 0.009, Si 0.0036, Cr 0.02, Ni 0.08, Cu $<$ 0.003, S 0.008, and P 0.0025.

The total content of nitrogen and carbon, estimated from the height of the internal friction peak at 30°C, was $\sim$ 0.001 percent by weight.

The A iron was annealed in wet hydrogen for 100 hours at 750°C to remove the carbon and nitrogen. The interstitial atoms in the B iron specimen thus obtained were less than 0.0002 percent by weight (estimated from the height of the internal friction peak).

A relaxer, designed and built under the supervision of Yu. V. Piguzov at the Moscow Institute of Steel and Alloys (MISS), was used to measure the internal friction. The relaxer is of the reverse torsion pendulum type. The /218
specimens were wire, 0.75 mm in diameter, with the working part 50 mm long. The internal friction was measured in the hardening experiment at -55°C, with the temperature created by pumping liquid nitrogen vapor through a coil. The low temperature made possible measurements at a distance from the Snoyek peaks, and this increased the accuracy of the measurements. Moreover, practically all aging processes in the hardened metal were brought to a halt during the measurements.

The specimen was in a vacuum of $\sim 1 \cdot 10^{-1}$ mm Hg. A constant magnetic field of $\sim$ 120 Oe was applied to reduce the magnetomechanical internal friction along the axis of the specimen.

Amplitude dependence was reduced in the $(2\text{-}30) \cdot 10^{-5}$ range. Measurements from low to high amplitudes were made at a frequency of 1.2 Hz. Internal friction at temperatures higher than ambient were measured in a vacuum of $1 \cdot 10^{-3}$ mm Hg. Temperature was maintained with an accuracy of ±1°.

Mechanical properties under tension were measured on a tensile strength tester of MISS design. The working part of the specimen measured 0.75 x 50 mm. The increase in the deformation on the diagram was 50. Accuracy of load determination was ~ 0.2 kg. Stress-strain curves were plotted for ambient temperature. The deformation rate was 6.4 percent/min. From 4 to 9 specimens were used for one point.

A vertical vacuum hardening furnace was used to heat-treat the wire specimens. The vacuum was $2 \cdot 10^{-3}$ mm Hg. Temperature was controlled and measured within ±5°C. The specimen was dropped into the hot zone of the furnace and kept there for 15 minutes. One minute before the end of the period, air was admitted to the furnace, and the specimen was dropped through the open lower part of the furnance into a salt water bath. Strict account was kept of the time the specimens remained at ambient temperature. Specimens were stored in a Dewar flask containing liquid nitrogen, when necessary.

All specimens were subjected to preliminary annealing at 850°C for 1 hour, using oven heating and cooling.

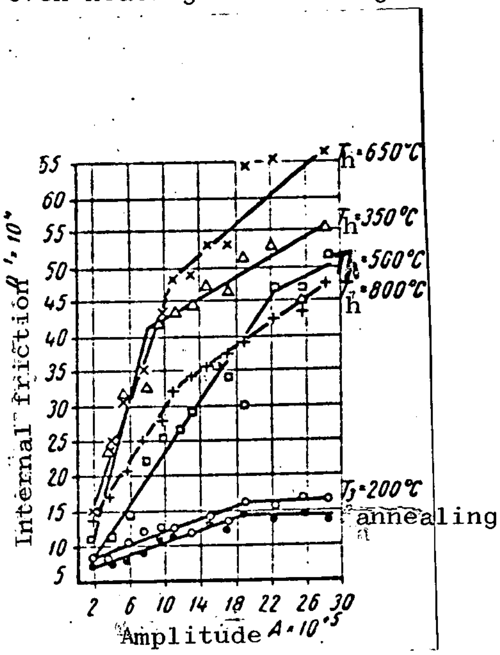

Figure 1. The effect of the hardening temperature on internal friction in a B iron specimen.

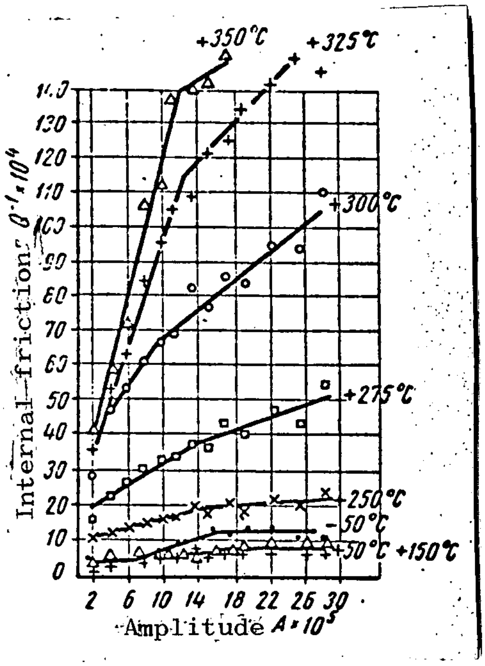

Figure 2. The effect of measurement temperature on internal friction in a B iron specimen.

Figure 1 shows internal friction in a B iron specimen in terms of amplitude when hardened successively at 200, 350, 500, 650, and 800°C. The internal friction was measured at -55°C. The elapsed time from hardening to the beginning of cooling in the relaxer was 1 minute. The figure shows that the amplitude dependence of the internal friction consists of two straight line sections, with the slope of the second section to the amplitude axis gentler than that of the first. This same relationship was observed in all the other cases.

Figure 1 further shows a sharp increase in the slope of the first section to the amplitude axis during hardening from 350-800°C. This indicates the release of the dislocations from the impurity atom "atmospheres." The hardening experiment data thus show that the dissipation of the "atomospheres" in B iron take place between 200 and 350°C and that rapid cooling makes it possible to retain unlocked dislocations in the metal. The scatter for curves in 350-800°C range is such that the change in the concentration of impurity atoms at dislocations in this range cannot be established, except to note that as compared to the annealed state and hardening at 200°C, the dislocations are unlocked. The picture is similar for all other specimens; a sharp increase in the slope between 200°C and 350°C, and broad scattering for curves between 350°C and 800°C. /219

Figure 2 shows the curves for internal friction in a B iron specimen in terms of amplitude, plotted for temperatures from -50°C to +350°C. Note that there is practically no effect on the amplitude dependence between -50°C and +150°C, but that there is a sharp increase in the slopes of the curves with increase in temperature, and in the overall level of internal friction, beginning at 250°C. This increase in the slope is associated with the dissipation of the impurity atom "atomspheres" at the dislocations above 250°C.

The quantitative processing of the curves in Figure 2 assumed that the magnitude $1/\alpha$, where α is the slope of the first section of the amplitude dependence to the amplitude axis, was proportional to the concentration of impurity atoms at the dislocations, c, and that Cottrell's formula [1] for the concentration of impurity atoms, c, at the dislocations, which follows, is valid in the region in which dissipation of the atmosphere takes place /220

$$c = c_0 \exp \frac{u}{kT}, \qquad (1)$$

wnere

c_0 is the concentration of the impurity in a solid solution;

u is the energy of the interaction between an impurity atom and a dislocation;

k is Boltzmann's constant;

T is absolute temperature.

According to Cottrell's calculations this formula is valid above the condensation temperature for the "atomspheres." But if, as Iouat and Beshers [4,5] say, the distribution of the impurity atoms near the dislocation is subordinate to Fermi statistics, then, and only then, should Eq. (1) be valid in that temperature range at which temperature begins to have a significant effect on this distribution.

What follows from these two assumptions is that log $(1/\alpha) = f\ (1/T)$ should be a straight line with slope u/k.

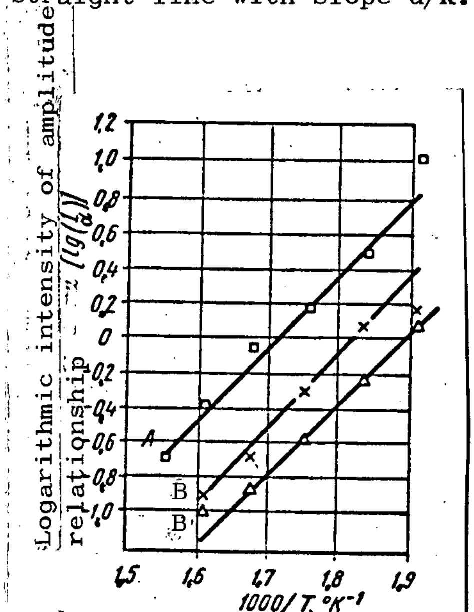

Figure 3. Influence of temperature on the intensity of the amplitude relationship for A and B iron specimens.

Figure 3 is a plot of log $(1/\alpha)$ values for two B iron specimens, and one A iron specimen. The points fall along a straight line quite well.

The slopes of the lines were used to calculate the process activation energy, which, obviously, can be identified with the energy of the interaction between nitrogen and carbon (in a mixture) and the dislocations in iron. It equals ~ 0.9 and 0.8 eV for the B specimens, and 0.9 eV for the A specimen.

Figure 3 further enables us to make yet another estimate of u. It can be pointed out that the $1/\alpha$ magnitude for the A iron specimen is reached at temperatures approximately 50°C higher than for the B iron specimens.

Substituting into Eq. (1) the concentrations for B iron, $C_0 = 0.0002\%$, and for A iron, $C_0 = 0.0010\%$, the temperatures 573°K and 623°K, and taking it that the concentrations of impurity atoms at the dislocations, C, at these temperatures /221 are equal for the A and B alloys, we obtain energy u = 0.9 eV, which agrees well with our previous estimate.

Thus, data from two methods used to study the internal friction show that in the 250-350°C range the "atmospheres" of impurity atoms are dissipated at the dislocations. This means we can obtain iron with dislocations locked by impurity atoms, as well as iron with dislocations that are not, and that we can investigate how the "atmospheres" influence the yield point.

Table 1 lists the mechanical properties of B iron after different hardenings. The specimen was at ambient temperature for approximately 3 minutes from hardening to the beginning of tension.

TABLE 1. MECHANICAL PROPERTIES OF B IRON AFTER HARDENING AT DIFFERENT TEMPERATURES

Hardening temperature, (T_h) °C	Proof stress, ($\sigma_{0.2}$), MN/m^2	Ultimate strength, (σ_u), (MN/m^2)	Elongation at end of yield stage, (ε_e), %	Total elongnation upon failure, (ε_Σ), %
annealing	74	118	1.3	17
200	74	118	1.0	13.5
250	74	122	0.6	15
300	70	118	0.6	15.5
350	66	118	0.1	16
500	66	118	0	14.5

The data in Table 1 show that there is a very great change in the magnitude of the yield stage with hardening temperature. It decreases from 1.3 percent to zero between 20°C and 350°C. In other words, no "atmospheres," no yield stage. The proof stress is unchanged, and the yield point has a tendency to decrease when the "atmospheres" are dissipated.

Impurity atoms should migrate to the dislocations, and once again lock them, when B iron specimens hardened at 350°C are aged at ambient temperature. This unique "deformation aging" takes place only in the case of dislocation densities corresponding to an annealed metal. Figure 4 is a graphic presentation of the change that takes place in the internal friction during such process, measured at -55°C. Table 2 lists the mechanical properties during tension. Figure 5 shows the change in the slope (for three specimens) of the first section of the internal friction-amplitude ratio, and of the deformation at the end of the yield stage with aging time. /222

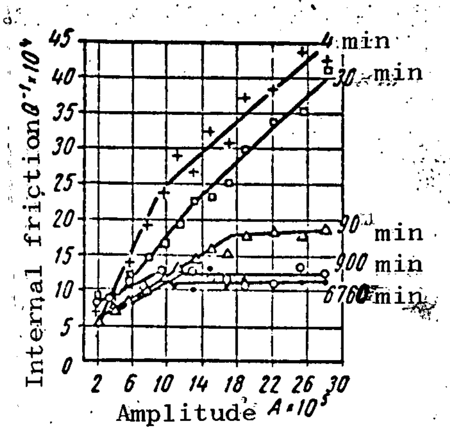

Figure 4. Effect of time at ambient temperature on the internal friction for a hardened B iron specimen.

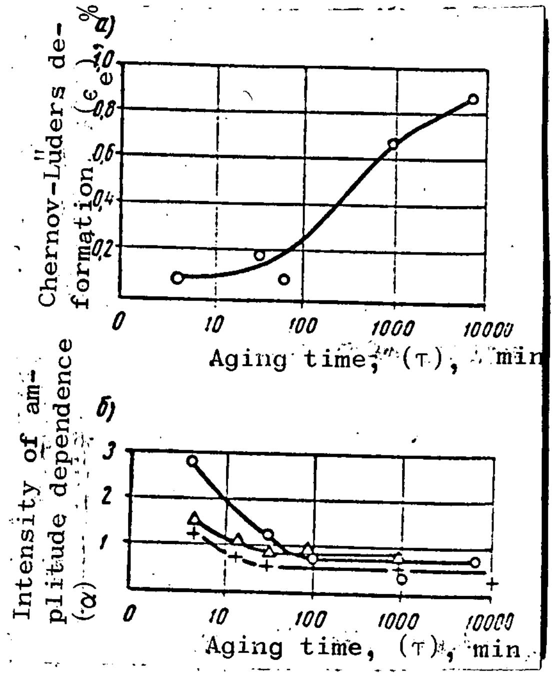

Figure 5. Change in B iron properties during aging. a - Chernov-Lüders deformation; b - Intensity of amplitude dependence.

TABLE 2. MECHANICAL PROPERTIES OF B IRON AFTER HARDENING AT 350°C AND AGING AT AMBIENT TEMPERATURE

Aging time, (τ), min	Proof stress, ($\sigma_{0.2}$), MN/m^2	Ultimate strength, (σ_u), MN/m^2	Elongation at end of yield stage, (ϵ_e), %	Total elongation upon failure, (ϵ_Σ), %
3	66	118	0.1	16
30	72	122	0.2	16
60	66	118	0.1	16
900	68	115	0.7	17
6780	78	122	0.9	13

The process proceeds in two stages. The first stage, which lasts for 30-90 minutes, involves a sharp reduction in angle α, falling to some minimum. The second section of the amplitude dependence also decreases its slope to zero during this same time span. In other words, independence of the internal friction that occurs at amplitudes greater than those which have a significant dependence on amplitude, puts in an appearance. The magnitude of the yield stage remains near zero during the first stage. The yield stage appears during the second stage of the process, and the height of the amplitudinally dependent region of internal friction decreases somewhat. This reduction is important in this case because all measurements were made with one specimen, which was not removed from the relaxer clamps. /223

The dependence of internal friction on amplitude obtained in the work described clearly is outside the approach in the Swartz-Weertman model [6], although the behavior of the internal friction on the first section does not agree with this model. One can, however, make certain assumptions concerning the process involved in the locking of free dislocations as a hardened specimen ages. During the first stage it is probable that the nucleus of the "atmosphere" itself is formed, leading to a significant reduction in angle α. This is a comparatively rapid process, because few carbon and nitrogen atoms are required when the density of dislocations is 10^6 - 10^8 cm^{-2}. The amplitudinally independent section predicted by Swartz and Weertman exists in the second stage, and the external region of the "atmosphere" is formed. In this case the increase in the concentration of impurity atoms in the neighborhood of the dislocation should reduce the level of the second section, the level $\sim c^{-1/3}$ [6], something that is, in point of fact, observed. The appearance of the external zone of the atmosphere leads to the occurrence of the yield stage. What is completely unclear in the supposed explanation is why, even after the occurrence of the external zone of the "atmosphere" there is a section in which the internal friction is heavily dependent on the amplitude. Further experiments will be required in order to answer all these questions.

Conclusions

1. Impurity atom atmospheres at dislocations in iron with a concentration of 0.0002 percent interstitial atoms are dissipated in the temperature range from 200°C to 350°C.

2. Rapid cooling can retain in pure iron dislocations that are free of atmospheres.

3. The energy of the interaction between interstitial atoms and dislocations in iron is $\sim$ 0.8-0.9 eV.

4. Unlocking dislocations from the "atmospheres" result in the disappearance of the yield stage.

5. The process involved in locking dislocations in pure, undeformed iron, takes place in two stages.

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